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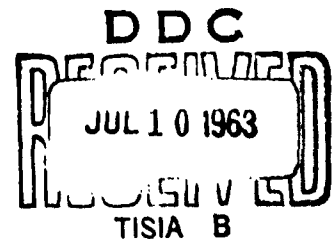
FEASIBILITY STUDY ON  
HEXAGONAL GLASS FILAMENTS

Final Report

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DeBell & Richardson, Inc.

ABSTRACT

It was demonstrated that hexagonal filaments can be made from E glass by the preform attenuation technique. A procedure was developed for grinding flats on round rods in order to produce hexagonal preforms. Strength tests on single filaments proved the desirability of acid polishing the ground preforms prior to forming filaments. Photomicrographs of a sample of hexagonal filaments forced together in an imbedding resin show perfect packing.

## INTRODUCTION

Earlier work at DeBell & Richardson, Inc., under a Bureau of Ships Contract<sup>1</sup> has shown that glass filament wound structures are subject to failure in shear in the non-glass region near the interface between the resin and the glass. It has been found in both laboratory and practice that up to a certain level, higher glass content yields higher strength of a filament wound structure.

It is our contention that this peak on the strength curve, at a glass content level where there should still be little glass-to-glass contact, exists because the packing has become so tight that the thin, almost line contact, zone between round filaments is subject to shear failure. The resin layer thickness varies so much around a filament in the region between two filaments that stress gradients become excessive leading to the shear failure.

Hexagonal glass fibers of regular, uniform cross section could provide the means for achieving high glass content as well as thin resin layers of large area for maximum stress transfer from one filament to the next.

At the conclusion of the program that DeBell & Richardson conducted for the Bureau of Weapons<sup>2</sup>, a brief exploratory study was made to show that flat sided glass filaments could be produced by the preform attenuation technique. Rectangular fibers made from narrow strips of window glass retained their sharp corners and flat sides down to a thickness of about 1/1000 of an inch. Further refinements in the technique have permitted us to produce fibers approximately the size of commercial round glass filaments and retain the flat sided shape of the preform. With this information in hand, we approached the Naval Research Laboratory with regard to an exploratory program for investigating the feasibility of producing filament wound structures from hexagonal filaments. It was the goal of this program to utilize the hexagonal shape to provide perfect packing of filaments into a honeycomb-like structure in cross section. The choice of the E glass composition for such a small program covering entirely new ground was, it turned out, an unfortunate one.

### TECHNICAL APPROACH

E glass rods were purchased from the Research & Development Division of Corning Glass Works at necessarily high cost and slow delivery. It was intended that we have these rods optically ground to form a hexagonal preform. This preform was then to be drawn into a continuous hexagonal filament with vapor phase application of A-1100 coupling agent and subsequently sprayed with a low viscosity epoxy resin as the filament wound on the mandrel. In this way, 12 hoops of the NOL type were to be made. The product of this program was to be photomicrographs of ground and polished slices from these filament wound structures and the determination of physical properties on the NOL type hoops made from E glass fibers with a hexagonal cross section.

It was intended that we contract to have the hexagonal rods ground. However, it was not possible to locate a concern to do this, therefore, we designed and constructed a machine for simultaneously grinding the two opposite flats on round E glass rods by passing the round rod between two thin diamond saw blades. Figure 1 shows a general view of this equipment. A bench grinder motor was adapted to operate the saw blades with suitable spacers and retainer discs. A steel table about twice the length of the rods was mounted firmly so that a  $120^\circ$  groove along its length would be centered on the gap between the two diamond saw blades. The glass rods were held with sliding clamps to prevent them from spiraling during grinding. By using a pair of clamps, it was possible to maintain the orientation of the rod by fastening the second clamp to the flats, formed by the diamond saws, after the shaped rod left the saws and, subsequently, removing the first clamp. Such a clamp can be seen in the close-up of the preform machine, Figure 2. This view shows the two closely spaced diamond saw blades and their back-up discs. After the first pass through the diamond saws, it was necessary to raise one-half of the table which was split along the bottom of the  $120^\circ$  groove, shimming it up about .020" to compensate for the fact that only one flat would be in the  $120^\circ$  groove during the second cut. Finally, for the third pass, the table was lowered to its original position. The rod on the right hand side of Figure 3 is an example of the product of this preform machine with its matte finish. Higher blade speed would probably minimize the chips from the corners. The end view demonstrates that the product of the machine can be a regular hexagon in cross section.

There are two distinct disadvantages to the use of E glass for this investigation. It has an extremely short working range requiring very precise temperature control, as well as steep thermal gradients within the fiber forming furnace. Another disadvantage of E glass is that it devitrifies readily at temperatures approaching those required for forming by the preform attenuation technique.

When we did achieve conditions which would produce hexagonal fibers, it was found that the strength of single filaments was quite low, approximately 50,000 psi. Some imperfections could be seen on the surfaces of these filaments and microscopic examination of the area being attenuated showed that the grinding imperfections were being carried through the filament forming process to the filaments themselves. Next, some hexagonal rods were tediously hand polished until only small microscopic scratch lines showed. However, fibers made from these exhibited only slightly improved strength. F. M. Ernsberger<sup>3</sup> of Pittsburgh Plate Glass Co. has shown that polished plate glass is covered with micro-cracks which supports our finding that mechanically polished hexagonal rods still produced weak fibers.

Since no particular strength increase accompanies mechanical polishing, it was decided to investigate acid polishing. Following the lead of Proctor<sup>4</sup> as well as Outwater and Ozaltin<sup>5</sup> who have had success in strengthening glass rods by etching or polishing in various acid mixtures to eliminate or minimize the effect of surface flaws, we made a study of the effect of exposing the ground E glass rods to various concentrations of hydrofluoric acid (HF) by itself and in combination with sulfuric ( $H_2SO_4$ ), hydrochloric (HCl), nitric ( $HNO_3$ ) and phosphoric ( $H_3PO_4$ ) acids.

HF in moderately high concentration, 30 to 50%, with or without the other mineral acids, accomplished a substantial degree of etch. However, even with a fair amount of agitation, a tenacious deposit was developed on the etched surface which was removed only by gently scrubbing in water after removal from the acid. At the end of the program, we were in the process of setting up to vibrate the rod with ultrasonic energy while immersed in the acid to see if we could eliminate the deposit and bring about superior polishing. However, we have been advised that this technique will maintain or even accentuate the roughness. A physical scrubbing action is thought to be more desirable.

To our knowledge, none of the earlier experimenters has used the rod polishing technique as a means of subsequently producing high strength fibers. However, it would seem to follow logically that low flaw-count rods should produce higher strength fibers than untreated rods.

The acid polished hexagonal rod samples we made which still showed visible shallow etch pits appeared to polish themselves during attenuation as noted by microscopic observation of the fiber forming section of the rod and stronger fibers were produced. Tensile tests on glass fibers require particular care and significant data calls for many more samples than we were able to make on this abbreviated program.



The hexagonal preform on the left in Figure 3 is an example of one of our better acid polished preforms. First, it was washed in acetone and then polished in 30% hydrofluoric acid for 40 minutes with constant agitation. Although we were not able to produce enough hexagonal filament to make a filament wound sample such as an NOL ring, Figures 4, 5 and 6 do show photomicrographs of a section through a sample that was ground and polished to demonstrate the shapes of filaments we did make.

To produce these photomicrographs, a small sample was prepared by doubling and redoubling a hank of hexagonal filaments to further increase their number. This group of fibers was then dipped into resin and forced through the tip of a medicine dropper to squeeze them into close proximity. The whole specimen, medicine dropper and all, was then cured and the sample prepared by grinding and polishing a flat surface right through the smaller portion of the medicine dropper.

#### SUMMARY

1. E glass rods (1/4" diameter) were obtained from Corning Glass Works. DeBell & Richardson ground the round rods into hexagonal cross section since no outside source for this service could be found.
2. A continuous hexagonal filament was drawn as evidenced by the attached photomicrographs, Figures 4, 5 and 6. No NOL type 6" rings have been made to date with hexagonal filaments.
3. The photomicrographs were made of a hand-assembled sample rather than from a slice of a ring. Hundreds of microscopic examinations have been made of samples to determine their cross sectional shape and size.
4. Some preliminary tensile tests on individual filaments have been made mainly in conjunction with the acid polishing to minimize flaws and increase strength. All the other physical properties would require finished hoops.

### CONCLUSIONS

1. It is possible to make hexagonal filaments of E glass by the preform attenuation technique. Some hexagonal filaments which measured .0007" or less across the flats were made.
2. Since no NOL type hoops were made, properties of filament wound structures were not measured.
3. E glass is a difficult composition to work with in the preform attenuation process when fibers with a cross section other than round are desired.
4. Further work in this area should include studies of various glass compositions such as Pyrex and some of the more chemically durable alkali glasses with long working ranges.
5. The continuation of this effort to make filament wound structures from hexagonal filaments should provide samples upon which physical properties can be measured to verify the premise that the thin flat resin layers between filaments would transfer greater loads and that higher glass contents would be possible with increased modulus.

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FIG. 1

Preform machine for simultaneously grinding two flats on round E glass rods. Guard and coolant nozzles have been removed for photograph.

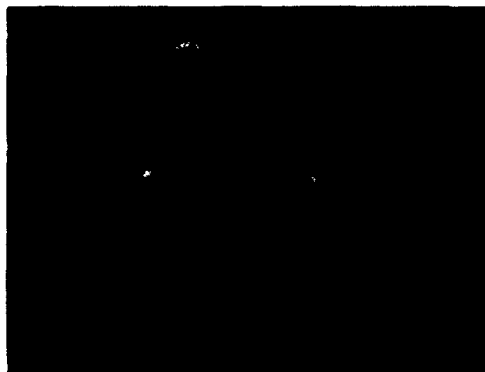


FIG. 2

Close-up of preform machine showing two thin diamond saw blades mounted on one shaft with gap between them centered on 120° groove in table. Hexagonal rod, previously ground, is gripped in clamp at right to keep rod from spiralling during grinding.

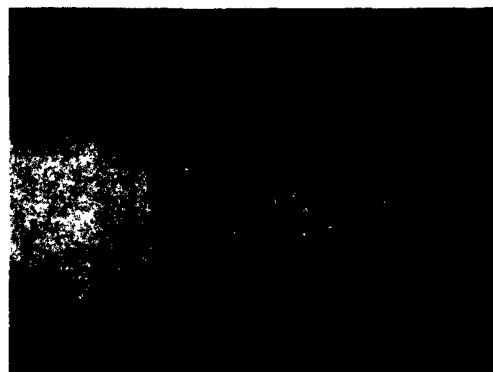


FIG. 3

Finished preforms. Rod on right has matte finish as ground by diamond saws. Left rod has been acid polished which yields a "greasy" surface.



FIG. 4

Photomicrograph (200X) of cross section through experimental hexagonal filaments. Note nearly perfect packing of center seven filaments.

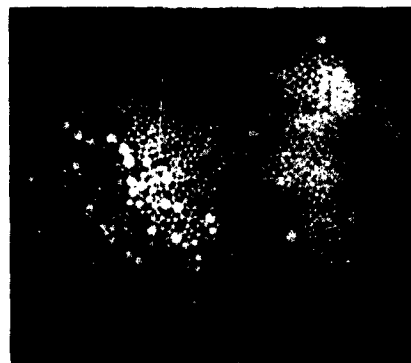


FIG. 5

General view (45X) of same sample made by squeezing fibers together. Several areas of dense packing are shown. The large filaments are from start-up.



FIG. 6

Another 200X view. Twelve filaments in the indicated area show good packing.